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भारतीय मानक

कार्बोक्सीन, तकनीकी — विशिष्टि

Indian Standard

CARBOXIN, TECHNICAL — SPECIFICATION

UDC 632·952 CARB

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

May 1991 **Price Group 2**

AMENDMENT NO. 1 DECEMBER 1999
TO
IS 13110 : 1991 CARBOXIN, TECHNICAL — SPECIFICATION

[*Page* 1, *Table* 1, *Sl No.* (ii), *col* 2] — Substitute the following for the existing matter:

'Melting point —89-92 °C'.

(FAD 1)

Reprography Unit, BIS, New Delhi, India

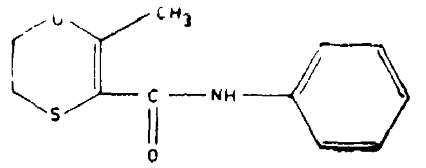
Pesticides Sectional Committee, FADC 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Division Council.

Carboxin, technical is used in the preparation of fungicidal formulations.

Carboxin is the accepted common name by the International Organization for Standardization (ISO) for 5, 6 dihydro-2-methyl-1, 4-oxathi-in-3-carboxanilide. The empirical and structural formulae and the molecular mass of carboxin are given below:

Empirical Formula	*Structural Formula*	*Molecular Mass*
$C_{12}H_{13}NO_2S$		235·3

In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act*, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard
CARBOXIN, TECHNICAL — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for carboxin, technical.

2 REFERENCES

The Indian Standards listed in Annex A are necessary adjuncts to this standard.

3 REQUIREMENTS

3.1 Description

The material shall be in the form of a white to pale yellow crystalline powder, free from extraneous impurities.

3.2 The material shall also comply with the requirements specified in Table 1.

Table 1 Requirements for Carboxin, Technical

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex of this Standard	Cl No. of IS 6940 : 1982
(1)	(2)	(3)	(4)	(5)
i)	Carboxin contents, percent by mass, *Min*	97·0	B	—
ii)	Melting point °C, *Min*	91	—	6
iii)	Acidity (as H_2SO_4), percent by mass, *Max*	0·2	—	11.3.2
	or			
iv)	Alkalinity (as NaOH), percent by mass, *Max*	0·1	—	11.3.3

4 PACKING

The material shall be packed in fibreboard drums provided internally with low density polyethylene lining of thickness not less than 0·062 mm. The drums shall have mild steel jacketed circular rim on top only. The locking device shall be provided with circular mild steel jacket. The containers shall also comply with the general requirements specified in IS 8190 (Part 1) : 1988.

5 MARKING

5.1 The container shall bear legibly and indelibly the following information and any other information as is necessary under the *Insecticides Act*, 1968 and Rules framed thereunder:

a) Name of the material;

b) Indication of the source of manufacture;

c) Date of manufacture and date of expiry;

d) Batch number;

e) Net mass of contents;

f) Carboxin content, percent (*m/m*); and

g) A cautionary notice as worded under the *Insecticides Act*, 1968 and Rules framed thereunder.

5.2 The containers may also be marked with the Standard Mark.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in IS 10946 : 1984.

7 TESTS

7.1 Tests shall be carried out by the methods referred to in col 4 and 5 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

8 PRECAUTION FOR HANDLING

Carboxin is an eye irritant and may cause itching to the skin. Rubber gloves shall be worn while handling the material.

ANNEX A

(*Clause* 2)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*
1070 : 1977	Water for general laboratory use (*second revision*)
6940 : 1982	Methods of test for pesticides and their formulations (*first revision*)
8190 (Part 1) : 1988	Requirements of packing of pesticides: Part 1 Solid pesticides (*second revision*)
10946 : 1984	Methods of sampling for technical grade pesticides

ANNEX B

[*Table* 1, *Item* (*i*)]

DETERMINATION OF CARBOXIN CONTENT

B-0 GENERAL

Two methods, namely, volumetric method and thin layer chromatographic (TLC) method can be used for the determination of carboxin content. However, TLC method shall be the referee method.

B-1 VOLUMETRIC METHOD

B-1.1 Principle

Carboxin is oxidized by hydrogen peroxide to its sulphoxide and the excess hydrogen peroxide is back titrated with standard sodium thiosulphate solution.

B-1.2 Reagents

B-1.2.1 *Hydrogen Peroxide Solution* — 15 percent (*v/v*).

B-1.2.2 *Potassium Iodide Solution* — 50 percent (*m/v*).

B-1.2.3 *Sulphuric Acid* — 3 N.

B-1.2.4 *Glacial Acetic Acid*

B-1.2.5 *Starch Indicator Solution*

B-1.2.6 *Standard Sodium Thiosulphate Solution* — 0·5 N.

B-1.3 Procedure

Weigh accurately a quantity of sample containing 1 0 g of carboxin into a 250-ml Erlenmeyer flask, add 20 ml glacial acetic acid and warm the contents of the flask on a steam-bath. Cool, add 2 ml hydrogen peroxide with the help of pipette and swirl to mix the contents. Cover the flask and allow to stand for one hour at room temperature. Add 10 ml sulphuric acid and then 10 ml potassium iodide solution. Titrate with standard sodium thiosulphate solution using starch as an indicator. Carry out a blank determination.

NOTE — For a sample of about 1 0 g, titres run is between 20 and 30 ml and the blank between 40 and 45 ml.

B-1.4 Calculation

Carboxin content, percent by mass $= \dfrac{(V_1 - V) \times N \times 1\,175}{M}$

where

V_1 = volume, in ml, of standard sodium thiosulphate solution required for blank determination;

V = volume, in ml, of standard sodium thiosulphate solution required for the titration of the sample solution;

N = normality of sodium thiosulphate solution; and

M = mass, in g, of the sample taken for the test.

B-2 THIN LAYER CHROMATOGRAPHIC METHOD

B-2.1 Principle

Carboxin is dissolved in chloroform This solution is subjected to thin layer chromatography and the carboxin zone is scraped from the developed plate. Carboxin is then eluted from the absorbent with chloroform and estimated spectrophotometrically at 300 nm (*see* Note)

NOTE — Carboxin is extracted from the formulations with chloroform. The extract is subjected to thin layer chromatography as in case of carboxin, technical.

B-2.2 Apparatus

B-2.2.1 *Ultra-Violet Spectrophotometer* — recording between 250 and 400 nm with 1 cm quartz cells.

B-2.2.2 *Thin Layer Chromatography Plates* — 200 mm × 200 mm.

B-2.2.3 *Air-Oven*

B-2.2.4 *Chromatographic Tank*

B-2.2.5 *Kuderna Danish Evaporator or Equivalent*

B-2.2.6 *Soxhlet Apparatus*

B-2.2.7 *Microlitre Pipette*

B-2.2.8 *Chromatography Micro-column* — 10 cm × 0·5 cm i.d., constricted to 2 mm bore at lower end.

B-2.3 Reagents

B-2.3.1 *Silica Gel* — TLC grade.

B-2.3.2 *Carboxin Reference Standard* — 99·8 percent plus purity.

B-2.3.3 *Chloroform* — spectroscopic grade.

B-2.3.4 *Calcium Chloride* — anhydrous.

B-2.3.5 *Potassium Permanganate Solution*

Dissolve 200 mg potassium permanganate in 10 ml water and add 5 ml sulphuric acid.

B-2.3.6 *Toluene*

B-2.4 Procedure

B-2.4.1 *Preparation of Carboxin Standard Solution*

Weigh accurately 0·100±0·001 g carboxin standard in a 100-ml volumetric flask, add 50 ml chloroform and swirl until complete dissolution of the material. Dilute the volume to the mark with chloroform.

B-2.4.2 *Preparation of Sample Solution*

B-2.4.2.1 *For carboxin, technical*

Weigh accurately a quantity of sample to contain 0·100±0·001 g carboxin in a 100-ml volumetric flask, add 50 ml chloroform and swirl until complete dissolution of the material and dilute to mark with chloroform.

B-2.4.2.2 *For carboxin formulations (Soxhlet extraction)*

Weigh accurately a quantity of sample to contain 0·100±0·001 g carboxin and transfer to the extraction thimble. Add 1 or 2 boiling stones and about 50 ml chloroform to the flask. Extract for 2 hours refluxing at a rate sufficient to complete extraction and later allow the solution to cool to room temperature. Dilute the cooled solution to 100-ml with chloroform.

B-2.4.3 *Chromatography*

B-2.4.3.1 Mix 40 g silica gel with 80 ml water shaking vigorously for 2-3 seconds. Spread 1 mm layer of the slurry over five plates and allow to air-dry for 1 hour before drying for another 1 hour in an oven maintained at 100°C. Remove the plates and store in a desiccator.

B-2.4.3.2 Presaturate the chromatographic tank with chloroform-toluene mixture [9 : 1 : (v/v)] for about 1 hour before use. Spot 100 µl each of sample and standard solutions about 3·8 cm apart on the plate and on side of the plate respectively. Air-dry the plate and place in the tank for development. Remove the plate after development from the chamber, air-dry and partially cover with glass plate, leaving the standard spot exposed. Spray potassium permanganate reagent on the plate to locate the spots. Scrap off corresponding spots of samples and standard separately and transfer to two different micro-columns. Elute carboxin with 50 ml chloroform and collect in 50-ml volumetric flasks. Dilute the volumes to the marks with chloroform and then concentrate to 10 ml in evaporator.

B-2.4.4 *Estimation*

Measure the absorbances of standard and sample solutions in a spectrophotometer at 300 nm using chloroform as blank.

B-2.5 Calculation

$$\text{Carboxin content, percent by mass} = \frac{A_1 \times M_2}{A_2 \times M_1} \times P$$

where

A_1 = absorbance of carboxin in sample solution;

M_2 = mass, in g, of carboxin reference standard;

P = percent purity of carboxin reference standard;

A_2 = absorbance of carboxin in standard solution; and

M_1 = mass in g of sample taken for the test.

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